

PATENT SPECIFICATION



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COMPLETE SPECIFICATION

Improvements in or relating to the Coating of Electrical Conductors.

We, STANDARD TELEPHONES AND CABLES LIMITED, a British Company, of Connaught House, 63, Aldwych, London, W.C.2, England, Assignees of CALVIN SOUTHER FULLER, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention relates to improvements in the coating of electrical conductors insulated with linear polyamides.

Linear polyamides have been heretofore disclosed, for instance, in British Specifications 461,236 and 461,237. Such polyamides are produced by condensation of suitable bifunctional amide-forming reactants which will react to form linear molecules. Thus, such a polyamide may be formed by condensing a single monocarboxylic monoamino acid, by condensing two or more of such acids in the desired proportions, by condensing one or more dicarboxylic acids and one or more diamines in the proper proportions, or by condensing equivalents of such substances. The reaction is carried out under conditions such that linear molecules of high molecular weight are obtained.

These linear polyamides may be of different degrees of crystallinity, ranging from polyamides which are substantially amorphous or non-crystalline at room temperatures to those which are substantially crystalline at such temperatures. The degree of crystallinity depends upon the nature of the chemical repeating unit or units making up the linear molecules of the polyamide. Such polyamides may be classified as substantially crystalline polyamides, i.e. those which are predominantly crystalline at room temperatures and have fairly definite melting points substantially above room temperatures; and substantially non-crystalline polyamides, i.e. those which are predominantly or entirely amorphous at room temperatures. Linear polyamides which are substantially crystalline at room temperatures and which have fairly definite melting points substantially

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above room temperatures have properties such as toughness, flexibility and tensile strength rendering them useful for various purposes. It has, for example, been proposed to form such polyamides into filaments useful for textiles or other purposes, and to employ such polyamides in the formation of films or coatings. In general, the desirable properties of such crystalline linear polyamides are associated with fairly high molecular weights and particularly with molecular weights high enough to permit cold drawing of the polyamides.

When initially solidified, such a crystalline linear polyamide appears to consist of haphazardly disposed micro-crystals associated with amorphous material of the same chemical composition which serves as a matrix and lends toughness and flexibility to the polyamide. When such a polyamide is cold drawn or linearly stressed at room or even at somewhat elevated temperatures the crystals and molecules become oriented in the direction of stress, as a result of which the strength, toughness and elasticity of the polyamide in the direction of stress are improved.

Such crystalline linear polyamides are, however, readily fusible and when heated above their melting points soften or melt, becoming deformable and losing their desirable properties, such as strength and toughness. Moreover, since the melting points of such crystalline polyamides are often relatively low in comparison to temperatures to which it may be desirable to subject them in use, the disadvantage occurs that they cannot be employed under conditions where they are subjected to more than moderate temperatures, or else the polyamides will either melt or soften and deform.

If such crystalline linear polyamides are aged by being heated below their melting points, particularly for prolonged periods, they become brittle and lose their elasticity and flexibility. Often, indeed, such embrittlement and loss of elasticity and flexibility arise upon mere aging of the material at ordinary temperatures, par-

ticularly in sunlight. These undesirable effects apparently are caused by crystal growth, particularly under the influence of heat, into the amorphous regions of the polyamide structure so that eventually the solid polyamide becomes substantially entirely crystalline in structure and contains practically no amorphous material which can lend toughness and flexibility to the material.

Specification 503,376 claims an electrical conductor provided with insulation comprising one or more synthetic linear condensation polyamides in oriented or unoriented form. For example, such polyamides may be employed as films or coatings for conductors or as impregnants for textile or paper insulation on conductors. However, the above-mentioned tendencies toward embrittlement and reduction of flexibility of such polyamides on heating or aging provide considerable disadvantages when the polyamides are employed in electrical insulation, since such tendencies cause such polyamides to crack and lose adhesion to their supporting means, such as the wire, with a resultant peeling and loss of moisture-proofing and insulating properties. The fusibility of such materials may also be disadvantageous when they are employed in electrical insulation, since heating of a conductor during use may melt or cause deformation of the insulating polyamide to such a degree that it fails to perform its protective insulating function.

Linear polyamides which are substantially non-crystalline in nature or which have a low degree of crystallinity may be liquids or solids of the nature of glasses, at room temperatures. In general, the liquids heretofore have been found to solidify only with difficulty, if at all, and hence have been heretofore found to be of little or no use. The more solid polyamides of low crystallinity are usually brittle glasses, or tend to become brittle or aging at ordinary or elevated temperatures, wherefore their uses have been limited. Such polyamides all soften and eventually liquefy as their temperatures are raised. The temperatures at which such polyamides liquefy or harmfully soften frequently are not high. The uses to which such substantially non-crystalline polyamides may be put are therefore limited by these factors.

Such difficulties heretofore encountered with polyamides are overcome by the present invention.

According to one feature of the invention we provide an electrical conductor having an insulating coating that is tough, flexible and substantially infusible and consists of a linear poly-

amide of intrinsic viscosity at least 0.1 that has been heated in the presence of oxygen to a temperature above its melting point and lying between 125° C. and 450° C. (preferably between 250° and 450° C.) for a time sufficient to change its colour at least to a pronounced yellow but not sufficient to change its colour deeper than a reddish brown.

According to another feature of the invention a process of treating an electrical conductor comprises coating the said conductor with a linear polyamide of such molecular weight that its intrinsic viscosity is at least 0.1 and afterwards heating the coated conductor to a temperature between 125° C. and 450° C. in the presence of oxygen for a time sufficient to change the colour of the polyamide at least to a pronounced yellow but not sufficient to change its colour deeper than a reddish brown and thereby converting the polyamide into a tough, flexible substantially infusible material.

In the practice of the invention the time of heating is a matter of a few minutes at the most. In most cases, particularly in the heat-oxygen treatments of substantially crystalline polyamides in which relatively high temperatures are employed, the time during which the polyamide is subjected to the maximum heat is in the neighbourhood of one minute or less.

A measure of the intensity of the heat-oxygen treatment to which the polyamide is subjected according to the present invention is afforded by the colour of the treated polyamide. As the polyamide is subjected to treatment of increasing intensity, its colour changes to yellow, then to brown, then to red-brown, then to dark red, and finally to black. In the case of a polyamide which is originally white or light coloured, the structure of the polyamide is not changed sufficiently to provide improved characteristics according to the invention unless the polyamide has been subjected to a heat-oxygen treatment of sufficient intensity to change its colour to a pronounced yellow. It is still more advantageous from the standpoint of desirable properties to subject the polyamide to a heat-oxygen treatment until its colour is brown to reddish-brown. A black colour indicates that the polymer has been overtreated and that harmful decomposition has occurred.

Exposure of an insulating coating on a conductor, of a substantially crystalline linear polyamide, to heat in the presence of oxygen according to the present invention results in an increase in the toughness and flexibility of the polymer and increased resistance to, if not entire

elimination of, the tendency to become brittle and inflexible upon aging at ordinary temperatures or upon prolonged heating below the melting point. By such treatment, moreover, the material may be rendered substantially, if not entirely, infusible and insoluble, at least in ordinary solvents. The adherence of the polyamide to its supporting surface is also greatly improved. By heat-oxygen treatment according to the present invention it is also possible to obtain from substantially non-crystalline linear polyamides or linear polyamides of a low degree of crystallinity flexible, tough, rubber-like films or coatings. Such rubber-like materials are substantially infusible and insoluble. The improved coating material of the present invention, is highly flexible, tough, abrasion resistant, does not tend to become brittle or inflexible upon aging or heating, is substantially infusible and hence not deformable when exposed to heat, and has extremely good adhesion to the conductor covered with it.

The heat-oxygen treatment of linear polyamides is advantageously performed by subjecting a suitable polyamide to heat of suitable intensity and to gaseous oxygen while the polyamide is in the liquid state and in a mass of thin cross-section. The gaseous oxygen may advantageously be that of the air, and the polyamide may be in the liquid state either because it is at a temperature above its melting point or because it is dissolved or because of a combination of these factors.

According to another feature of the invention a process of coating an electrical conductor with insulating material comprises covering the said conductor with a thin coating of a liquefied linear polyamide of such molecular weight that its intrinsic viscosity is at least 0.1 and immediately thereafter passing the coated conductor through an oven in which it is heated in air to a temperature between 125° C. and 450° C., the time of passage being sufficient to change the colour of the said coating to a pronounced yellow but not sufficient to change its colour deeper than a reddish brown, thereby converting the polyamide into a tough, flexible substantially infusible material.

In the case of a polyamide which is a substantially crystalline solid at room temperatures, the temperature to which the polyamide is heated during the heat-oxygen treatment is above the melting point of the polyamide. Advantageously, it is considerably higher than the melting point. While the temperature of heating of such a substantially crystalline polyamide is determined by the melting point

and structure of the polyamide, the temperatures employed in the practice of the invention lie between 250° C. and 450° C., being in each case above the melting point of the polyamide being heated. From the standpoint of the desirable results obtained, it is particularly advantageous to employ temperatures lying between 325° C. and 450° C.

If the polyamide is of the non-crystalline type, i.e. is originally liquid or plastic at room temperatures, the heat should be of such intensity that the polyamide is transformed into a tough, rubber-like mass which is substantially infusible and insoluble. In this case, too, the intensity of the heat to which the polyamides should be subjected is dependent upon the structure of the polyamide, and in the practice of the present invention such polyamides are heated to temperatures lying between 125° C. and 450° C. until the desired structural changes occur.

A sufficient amount of oxygen, in general an excess, is employed to provide the desired changes in the structure of the polyamide at the heat intensity employed. When the polyamide is dissolved in a solvent and exposed to heat and the oxygen of the air, the solvent is evaporated by the heat and dilutes the oxygen in the air. In such a case sufficient air must be supplied to ensure an ample supply of oxygen for the reaction, particularly if the polyamide is heated in an enclosure, such as an oven. Usually at least one per cent. by volume of the atmosphere surrounding the polyamide should be oxygen for satisfactory results.

The most desirable results are obtained, according to the present invention, when the linear polyamides which are subjected to the heat-oxygen treatment have molecules which are of fairly high average molecular weights. The molecular weight of a polyamide may be expressed as a function of the intrinsic viscosity of the polymer, and it is necessary that the polyamides treated according to present invention have an intrinsic viscosity in a suitable solvent, such as *m*-cresol, of at least 0.1. The intrinsic viscosity may be determined from the formula:

$$V_i = \frac{\log_e V}{C} \quad 120$$

where *V* is the viscosity of a fairly dilute solution of the polyamide divided by the viscosity of the solvent in the same units and at the same temperature, and *C* is the concentration in grams of the polyamide per 100 cc. of the solution. Even better

results are obtained if the intrinsic viscosity of the material is above 0.4. In crystalline linear polyamides an intrinsic viscosity of this magnitude indicates a molecular weight at about which cold drawing begins to be possible.

The present invention relates to coating an electrical conductor with heat-oxygen treated saturated linear polyamides, i.e. those substantially free of non-benzenoid unsaturation. The linear molecules of such saturated polyamides may be of various forms. Thus, they may be straight aliphatic chains or contain side chain substituents connected to either of, or to both, the carbon and the nitrogen atoms. Such substituted polyamides, in general, are less crystalline than the unsubstituted polyamides. The linear molecules may contain in their chains hetero atoms, such as oxygen or sulfur atoms, with or without substituting groups. It is, however, advantageous to employ straight aliphatic chain polyamides since the improved polyamides of the present invention produced therefrom are of exceptional toughness, flexibility and aging resistance.

In forming an improved polyamide coating of the type contemplated by the invention by exposure to heat and gaseous oxygen of a liquified suitable linear polyamide coating, the thickness of the body of the polyamide thus exposed is important. In general, a thickness of not over about 1.64 of an inch should be employed and advantageous results are provided when the thickness is on the order of one mil or less.

The thickness of the body of the polyamide exposed to heat and oxygen is limited. Under the influence of the heat oxygen molecules penetrate the surface of the mass of the polyamide and cause changes in the molecular structure thereof. The portions of the polyamide nearest the surface are first affected and converted and subsequent oxygen molecules must penetrate such converted portions to reach the more inner portions. If the thickness of the body is too great, the oxygen molecules cannot penetrate into the innermost portions to completely treat the mass of the polyamide within a reasonable time and without decomposition of the outermost surfaces.

The desirable properties of linear polyamides which have been treated according to the present invention are due to the fact that the heat-oxygen treatment causes cross-linkages to occur between adjacent long-chain molecules thereof. It appears that most, if not all, of the cross-linkages are in the vicinity of the amide linkages of the molecules; that is, it appears

that in each of several amide linkages in a molecule either a nitrogen atom or a carbon atom adjacent thereto is linked to a nitrogen atom or to a carbon atom adjacent to a nitrogen atom in an adjacent long-chain molecule. The linkages are not amide linkages and contain no carbon atoms other than those in the linear molecules. The linkages are probably direct, but may be through oxygen. The long-chain molecules are thus connected together by cross-linkages to form a complex network, the number of such linkages being determined by the nature and intensity of the heat-oxygen treatment to which the material was exposed.

Since the molecules are thus interconnected, the material is substantially infusible and insoluble. For example, such a polyamide which was initially crystalline before treatment and which subsequent to treatment is still crystalline is opaque at room temperatures because of its crystallinity. As the temperature of the heat-oxygen treated polyamide is raised, no change occurs until a certain temperature is attained at which the polyamide becomes transparent. Upon subsequent cooling, it again becomes opaque. The change from opacity to transparency on heating occurs because the polyamide is no longer crystalline at the elevated temperature. Further heating of the polyamide in its transparent state does not cause it to melt or flow; indeed, if the heating is sufficiently intense, the polyamide will decompose without melting. An initially substantially non-crystalline polyamide which is converted according to the present invention into a tough, elastic mass will not melt upon being heated but if heated to a sufficient temperature will also decompose without melting.

Linear cross-linked polyamides are substantially insoluble in all common solvents. That is, although upon exposure to a solvent certain materials may in some cases be extracted from the polyamides, the mass of the polyamide is largely, if not entirely, unaffected, since the cross-linking of the molecules thereof prevents the disintegration into molecules necessary for a true solution.

A polyamide which, prior to treatment according to the present invention, is substantially crystalline at room temperatures still is largely crystalline after heat-oxygen treatment and, therefore, has the strength associated with the presence of crystallised linear molecules. The crystals, however, are small and are contained in a matrix of amorphous material, and the cross-linkages between the molecules tend to keep such crystals small.

Even prolonged heating of the kind which with ordinary crystalline linear polyamides causes embrittlement does not cause the crystals of the improved polyamide of the invention to increase in size and cause embrittlement, with a consequent loss of flexibility and toughness of the polyamide. The resistance to aging at ordinary temperatures is even more pronounced. When a crystalline polyamide is employed as a coating the cross-linking causes it to adhere tenaciously to its supporting material and even prolonged heating does not appreciably decrease such tenacity.

An initially substantially non-crystalline polyamide, or one of a low degree of crystallinity, which has been heat-oxygen treated according to the present invention is converted into a tough, rubber-like mass which is generally non-crystalline also. This apparently occurs because the cross-linkages between long-chain molecules cause gelation of the polyamide. Such improved polyamides may have some crystals therein, depending upon the tendency of the material to crystallize prior to treatment according to the invention. Such material also has good resistance to aging at both ordinary and elevated temperatures and, when employed as a coating or impregnant, has good adherence to its supporting substance.

During the heating and exposure to oxygen according to the present invention, some decomposition of the polyamide occurs with a resultant tendency to shorten the molecular chains. In a suitable linear polyamide suitably treated according to the invention, this tendency is offset by or is relatively minor in comparison to the tendency of the molecules to increase in molecular weight due to building up of the molecules by cross-linking. The intensity of the heat-oxygen treatment, therefore, should be such that the cross-linking action is favored but should not be so great that harmful decomposition occurs. Yet, as has been indicated, a heat-oxygen treatment of considerable intensity is required to cause the cross-linking to occur, since the invention relates to more than a mere drying of the polyamide.

The polyamide should be in the liquid state while being treated in order to facilitate the cross-linking of the molecules which would otherwise be impeded if, for example, the polyamide contained crystals.

Because of its toughness, flexibility, aging resistance, abrasion resistance and because of the tenacity with which it adheres to a material to which it has been

applied before being heat-oxygen treated, and its good electrical insulation properties, such polymeric insulating material is particularly advantageous. Thus, a bare metal conductor, such as wire, may be coated with a suitable linear polyamide according to a suitable conventional procedure for coating wires and then be advantageously exposed to the desired temperature and to oxygen, preferably before the polymer has had an opportunity to solidify. A continuous process may be very advantageously practiced according to which the conductor, such as wire, is continuously coated with a liquefied polymer after which it is continuously passed into a suitable heating oven which heats it in air. One or more coatings may be readily applied to a wire in this manner. High speeds of coating may be obtained with resultant economies in manufacture.

The preparation of linear polyamides of suitable molecular weights to be applied as coatings for electrical conductors and for subsequent treatment according to the present invention may be accomplished in any suitable manner. If the polymers are prepared by condensation of a diamine and dicarboxylic acid, or by condensation of two other bifunctional reactants having different reactive groups at the ends of the molecules, equimolecular or substantially equimolecular proportions should be employed to obtain long-chain molecules. When the polymer is prepared by condensation of two or more amino acids or the like, any proportions may be employed. The reactants are ordinarily heated in a suitable container, such as a glass reaction vessel, under conditions such that the condensation is promoted. For example, removal of water vapour or other condensation by-products to promote the condensation may be accomplished by continuously bubbling a dry oxygen-free gas, such as nitrogen or hydrogen, through the reaction mixture with or without application of reduced pressure. Alternatively, the water vapour may be removed by agitating the mixture at a reduced pressure so as to produce a greatly extended surface from which the condensation products can be removed. These or other expedients are required in order to attain molecular weights of the polyamide of the desired magnitude within a reasonable time. The presence of oxygen in the initial polymer-forming stage is undesirable since it will prevent attainment of the desired molecular weights. When the substance has reached the desired molecular weight, as is evidenced by a check of its intrinsic viscosity or by other means, it may be employed for forming films such

as coatings or the like, which may be subsequently treated according to the present invention.

5 In each of the following examples the polyamide is heated for a time sufficient to change its colour to a pronounced yellow but not sufficient to change its colour deeper than a reddish brown and is thereby
10 converted into a tough, flexible substantially infusible material.

EXAMPLE 1.

Equimolecular proportions of hexamethylene diamine and adipic acid were heated together in a glass reaction vessel
15 in the absence of oxygen to a temperature of approximately 270° C. for about 16 hours. A stream of oxygen-free hydrogen gas was passed through the molten mixture during the heating to remove the water vapour formed. At the end of the
20 heating the polyhexamethylene adipamide had an intrinsic viscosity in *m*-cresol of approximately 0.8. When solidified, the material was a cream-coloured, micro-crystalline solid melting at approximately
25 260° C. About 15 grams of this material were dissolved in 100 cc. of cresol to provide a mobile solution of the polyamide suitable for application to copper wire. By
30 means of the applicator of a horizontal wire enamelling machine, operating on conventional principles, a coating of the polyamide was applied to No. 30AWG copper wire. The wire was passed
35 continuously through the applicator and then through an electrically heated oven approximately 6 feet long at a rate of about 30 feet per minute, the oven being heated to a temperature of about 400° C. A coating
40 of approximately 0.3 mil average thickness was obtained in 4 passes of the wire through the apparatus. An ample supply of oxygen was provided by a forced draught of air through the oven at about
45 6 cubic feet per hour.

The coated wire had a cherry red colour and was extremely tough and abrasion resistant. The coating was crystalline and
50 sufficiently flexible to permit the wire to be bent around a mandrel of its own diameter without cracking, indicating that it had an elongation of at least 50 per cent. The hardness and abrasion resistance of the coated wire were compared with those
55 of a standard sample of No. 30 AWG varnish enamel wire and were found to be greatly superior.

When under the same operating conditions air was excluded from the oven
60 and nitrogen gas was passed therethrough, a coated wire was obtained the coating of which was colourless, very brittle, and very poorly adherent to the wire.

EXAMPLE 2.

65 In this example polyhexamethylene

adipamide, prepared according to the procedure outlined in example 1, of an intrinsic viscosity in cresol of 0.9 was dissolved in cresol to form a 20 per cent. solution. The solution was diluted with 13
70 per cent. by weight of high-flash naphtha. The resulting solution was employed in the apparatus of the type outlined in example 1 to coat No. 30 AWG copper wire. The wire speed was 40.8 feet per
75 minute and the oven temperature was 330° C., a coating of 0.65 mil thickness being obtained in 5 passes. The coating of the completed wire was of light brown colour, showed greater than 50 per cent. elongation and had a static breakdown of
80 2770 volts per mil (RMS). The abrasion resistance of the wire was tested by means of a sapphire having an edge of 3 mil radius of curvature which was pressed with increasing force against the coated wire as it was drawn past the edge, until the edge of the sapphire pierced the coating. The
85 "scraper value" of the coated wire or pressure to cause failure of the coating was 385 grams per mil of coating thickness, while well baked varnish enamel wire of the same gauge and thickness has a scraper value of only 200 to 250 grams per mil. The heat-oxygen treated polyamide coating on the wire was crystalline. It was
90 very tough and showed great adherence to the wire. The excellent ageing characteristics of the coating were shown by the fact that after being heated in air at 120° for 12 days the coating still had more than
95 50 per cent. elongation, while well processed varnished enamel wire drops from its initially low 35 per cent. elongation to less than 16 per cent. elongation in the
100 same time under the same conditions.

EXAMPLE 3.

Hexamethylene diamine and sebacic acid in substantially equimolecular proportions were heated together in a glass
110 reaction vessel at about 250° C. for approximately 16 hours, oxygen-free hydrogen being bubbled through the mass during the reaction to remove the water vapour formed. At the end of this time
115 the material had a sufficiently high molecular weight to permit the molten mass to be formed into threads which could be cold drawn into oriented fibres. When the mass, polyhexamethylene sebacamide, was solidified after heating, it was a white, micro crystalline solid having a melting
120 point of approximately 210° C. A solution of this compound was made by dissolving 40 grams in cresol and diluting the resulting solution with an equal volume of high-flash naphtha. The solution was employed to coat No. 40 AWG
125 copper wire by means of the horizontal enamelling machine described in example 130

1. The wire was continuously passed through the applicator and oven at about 71 feet per minute, the oven temperature being 250° C. and a forced draught of air of about 6 cubic feet per hour being passed through the oven. In 4 passes of the wire through the apparatus, the diameter of the wire was increased by the coating by 0.3 mil. The finished coating film was crystalline in character. The coated wire was tested in mercury under 5 volts potential and showed ten defects per 100 feet, which is relatively few for such an extremely thin coating of insulation. The scraper value for this wire was much higher than that for similar wire coated with a varnish enamel coating of like thickness. The coated wire showed an elongation greater than 50 per cent when tested by the mandrel test described in example 1. It was also found possible to solder this wire without scraping the insulation therefrom, as is necessary with other types of enamelled wire.

EXAMPLE 4.

Substantially equimolecular proportions of decamethylene diamine and sebacic acid were heated together in a glass vessel at about 250° C. with oxygen-free nitrogen being bubbled through the mass to remove the water vapour formed. After about 16 hours heating the mass was found to have an intrinsic viscosity of about 0.5 in cresol and after solidification was a white, microcrystalline solid melting sharply at about 195° C. This material, polydecamethylene sebacamide, was applied in its molten condition to No. 22 AWG tinned copper wire by means of enamelling apparatus of the character described in example 1. A 29 mil diameter die was employed to strip off excess of the melted polyamide. The molten polyamide in the apparatus was maintained in a closed reservoir under nitrogen at 250° C. —260° C. The coating of the polyamide of approximately 2 mils thickness was allowed to solidify in air on the wire, which was then coiled up. The wire was subsequently subjected to a temperature of about 450° C. in air for about one minute while it was maintained in a vertical position. The coating was converted by this treatment into a substantially infusible, insoluble, crystalline, brownish film. The wire so treated was tested in comparison with a like portion of the untreated wire by ageing in an oven at 130° C. for 24 hours. The untreated wire become excessively brittle losing adhesion to the conductor even upon slight bending, whereas the heat-oxygen treated coating adhered tenaciously to the wire even though it was bent around a mandrel of its own diameter. The toughness and

abrasion resistance of the coating were also extremely good.

EXAMPLE 5.

Sebacic acid and ethylene diamine were reacted in alcohol solution until a neutral reaction was obtained with bromo-thymol-blue, the purified salt resulting from the reaction being heated at about 250° C. in a stream of inert oxygen-free gas until the intrinsic viscosity in cresol was approximately 0.3. The polyethylene sebacamide so formed was a hard, brittle microcrystalline solid melting at about 260° C. A solution of 10 grams of this material in 50 cc. of cresol was employed to form a film about 0.5 mil thick on copper foil, the film being heated in air at about 300° C. for about one minute. A dark red, flexible, tough, adherent, infusible crystalline coating was obtained. A sample of the coated material was tested by immersion in water at room temperature for adhesion and solubility. Immersion for over 3 weeks caused no significant change in properties.

EXAMPLE 6.

Omega-amino undecanoic acid was converted into a polyamide by heating it at a temperature of about 250° C. for 14 hours in a stream of hydrogen gas. The average molecular weight of the polyamide at this stage was sufficiently high to permit it to be cold drawn into strong oriented fibres. In this case, however, a film of the polyamide was formed on a steel plate by spreading the molten polyamide to a thickness of approximately 2 mils and heating it in air to about 250° C. for several minutes. A very tough, insoluble, infusible, adherent crystalline coating was obtained having good electrical insulating properties.

EXAMPLE 7.

A polyamide was formed by reacting at about 250° C. 7.2 grams of N,N¹ dimethyl hexamethylene diamine with 7.3 grams of adipic acid in a stream of hydrogen gas for 3 days. The poly N,N¹ dimethyl hexamethylene adipamide so obtained was a sticky, viscous liquid having a molecular weight sufficiently great to cause the polyamide to have an intrinsic viscosity in *m*-cresol of considerably over 0.3. A solution of 5 grams of the polyamide in 50 cc. of chloroform was employed to form on a copper foil a thin film 0.5 mil thick. The film was heated in air at a temperature between 250° C. and 350° C. for 2 minutes. A rubber-like, infusible, tough, substantially non-crystalline film which adhered strongly to the metal foil was formed. When tested in chloroform the film proved to be insoluble.

EXAMPLE 8.

Poly N,N¹ dimethyl hexamethylene

adipamide as prepared in the preceding example was dissolved in chloroform to form a 10 per cent solution by weight. A length of No. 22 AWG copper wire insulated with two servings of cotton yarn was impregnated with said solution. The impregnated wire was dried at 60° C. and subjected to a temperature of 130° C. for 10 minutes in air. The wire showed improved insulation resistance and abrasion resistance as compared with the untreated wire. A portion of the wire was tested for burning and proved considerably more flame resistant than the unimpregnated wire.

EXAMPLE 9.

One mol of sebacic acid, 0.5 mol of propylene diamine, and 0.55 mol of ethylene glycol were reacted at about 250° C. for 22 hours in a stream of inert oxygen-free gas. The polyesterpolyamide so obtained was hard and tough, and was capable of being drawn into strong oriented fibres, its intrinsic viscosity in cresol being in the neighbourhood of 0.4. Of the sum of the ester and amide linkages in the linear molecules in this polyester-polyamide approximately 50 per cent were amide linkages. A solution of this polymer was prepared by dissolving 20 grams of this substance in about 100 cc. of cresol and diluting to about 14 per cent solids with more cresol. The resulting solution was applied to No. 31 AWG copper wire in a continuous manner by means of the apparatus described in example 1. A speed of 25 feet per minute of the wire and an oven temperature of 320° C. were employed, air being supplied to the oven at the rate of about 6 cubic feet per hour. The resulting crystalline coating was 0.4 mil thick and was tough, flexible, abrasion resistant and of good adherence. The wire coated with this material could be bent around a mandrel of the diameter of the wire without cracking, indicating that the coating had an elongation greater than 50 per cent. It retained this elongation even on prolonged heating in the neighbourhood of 100° C.

As indicated by the above examples, the polyamide may be formed into a coating, either from a solution or from the molten state. When initially subjected to the heat the polyamide may be in the liquid state or it may be in the solid state and be liquified by the heat, so that in either case the liquid state desirable for efficient heat-oxygen treatment is present. In any case where it is desired to convert the entire polyamide coating into an infusible, insoluble mass by exposure to heat and gaseous oxygen, care should be taken that the thickness of the polyamide is not too great to prevent penetration of the oxygen

to the innermost portions of the polyamide.

In the present invention, driers such as cobalt resinate, lead linoleate or cobalt naphthenate may be added to the polyamide or to the solution of the polyamide to accelerate the heat-oxygen treatment.

As has been indicated by example 9, the invention may be employed not only in connection with polymers the molecules of which contain in the chain only amide linkages between the repeating units, but also in connection with polymers the molecules of which contain in the chain other kinds of linkages besides amide linkages. At least 50 per cent of the molecular chain linkages, however, should be amide linkages in order that the cross-linking in the vicinity of the amide linkages which provide the desirable properties of the materials in the present invention may occur. The number and kind of linkages other than the amide linkages in the molecules should be such that they do not cause harmful degradation or decomposition of the polymer on being subjected to the heat-oxygen treatment. In the appended claims the term "polyamide" is intended to include, besides polymers containing only amide linkages, polymers containing other linkages besides amide linkages, provided that at least 50% of the molecular chain linkages are amide linkages.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

1. An electrical conductor having an insulating coating that is tough, flexible and substantially infusible and consists of a linear polyamide of intrinsic viscosity at least 0.1 that has been heated in the presence of oxygen to a temperature above its melting point and lying between 125° C. and 450° C. (preferably between 250° C. and 450° C.) for a time sufficient to change its colour at least to a pronounced yellow but not sufficient to change its colour deeper than a reddish brown.

2. A process of treating an electrical conductor which comprises coating the said conductor with a linear polyamide of such molecular weight that its intrinsic viscosity is at least 0.1 and afterwards heating the coated conductor to a temperature between 125° C. and 450° C. in the presence of oxygen for a time sufficient to change the colour of the polyamide at least to a pronounced yellow but not sufficient to change its colour deeper than a reddish brown and thereby converting the polyamide into a tough, flexible substantially infusible material.

3. A process of coating an electrical

conductor with insulating material which comprises covering the said conductor with a thin coating of a liquefied linear polyamide of such molecular weight that
5 its intrinsic viscosity is at least 0.1 and immediately thereafter passing the coated conductor through an oven in which it is heated in air to a temperature between 125° C. and 450° C., the time of passage
10 being sufficient to change the colour of the said coating to a pronounced yellow but not sufficient to change its colour deeper than a reddish brown thereby converting the polyamide into a tough, flexible, sub-
15 stantially infusible material.

4. Process as claimed in Claim 2 or 3 in which the linear polyamide with which the conductor is originally coated is a crystalline solid at room temperature and
20 the coated conductor is heated to a temperature between 250° C. and 450° C.

5. Process as claimed in Claim 4 in

which the said temperature is between 325° C. and 450° C.

6. Process as claimed in Claim 3 in
25 which the polyamide is brought into the liquid state by dissolving it in a solvent.

7. Process as claimed in Claim 3 in which the polyamide is brought into the liquid state by heating it to above its
30 melting point.

8. The respective processes of treating an electrical conductor described in the examples 1 to 9.

9. An electrical conductor coated by
35 any of the processes claimed in Claim 8.

Dated this 27th day of March, A.D. 1942.

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